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EFFECTS OF METAL CONTAMINATION ON HYDROXYLAMMONIUM NITRATE-BASED LIQUID MONOPROPELLANTS

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13. ABSTRACT *(Maximum 200 words)*

A hydroxylammonium-nitrate-based liquid gun propellant, LGP 1846, was intentionally contaminated with four separate metals and subjected to storage, shock sensitivity, and closed-bomb tests. The metal contaminants used in this study were iron, copper, nickel, and aluminum. The contaminant concentrations for the storage and the sensitivity tests were approximately 25, 75, and 100 ppm. For the closed-bomb tests, a concentration of approximately 100 ppm was used. Results indicated that the contaminated samples were stable for at least two weeks. The addition of the contaminants increased the sensitivity of the LGP 1846 slightly. The closed-bomb results showed that the contaminants decreased the time required to ignite the propellant by hot wire. The closed-bomb tests also showed that the nickel and aluminum contaminants tended to decrease ignition delay times by 22% and 19.5%, respectively. The iron and copper samples showed 5.6% and 2.6% decreases in ignition delay. Also, the fizz-to-flame transition pressure was found to differ from that found in a previous study.

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TABLE OF CONTENTS

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LIST OF FIGURES

LIST OF TABLES

1. INTRODUCTION

Numerous studies have indicated that the presence of contaminants, particularly transition metals, adversely affect the properties of the hydroxylammonium-nitrate (HAN)-based monopropellants. A study by Fifer, et al.,[1] showed that low levels of copper and iron, under 100 ppm, significantly lower the thermal initiation temperature of LGP 1845, a HAN-based propellant. Also, the catalytic effect of iron on the propellant is considered to greatly decrease the storage life of the HAN-based liquid propellants.[2] Compounding the problem is the ease at which some metals dissolve into solution with the HAN.[3] For example, a study by Decker[3] reported a concentration of 49,000 ppm of nickel after only one week of contact with 13.25 molar HAN.

The purpose of this study was to evaluate the ballistic characteristics of the HAN-based liquid gun propellants after contamination with various metal ions. The four contaminants of interest in this study were iron, copper, nickel, and aluminum.

Three different experiments were performed on the samples: short-term storage tests, shock-sensitivity tests, and closed-bomb tests. The short-term storage tests were performed to determine if the contaminated propellant would react or be unsafe to handle during the test periods. The shock sensitivity tests were performed, using a drop-weight apparatus, to evaluate the relative sensitivity of the propellant to compression ignition in a partially filled chamber. Last, closed-bomb or constant-volume tests, adapted from a similar approach used with solid propellants, were used to evaluate the ignition and combustion characteristics of the contaminated liquid propellants.

2. APPROACH

The LGP 1846 was used in all tests. LGP 1846 is composed of 60.79 percent HAN as oxidizer, 19.19 percent triethanolammonium nitrate (TEAN) as fuel, and 20.02 percent water as diluent and moderator (all percent by weight). The density and impetus of LGP 1846 are 1.43 g/cm^3 and 898 J/G. LGP 1846 is formulated stoichiometrically, such that the final combustion products are water, carbon dioxide, and nitrogen.

Samples of LGP 1846 were contaminated with metal ions of copper, nickel, iron, and aluminum. Each metal was tested separately, and no metals were used in combination. The sulfate salts of the metals were weighed in quantities to produce approximately 25, 50, and 100 ppm of each of the metals being tested. Once the salt was added to the propellant, the solutions were agitated

agitated gently with a magnetic stirrer. Stirring was maintained without the introduction of air bubbles into the solutions. Gas bubbles were noticed in the iron samples before agitation was started. Once the salts were dissolved, the metal content of the solutions was verified by analysis with an inductively coupled plasma spectrophotometer.

2.1 Storage Tests. Short-term storage tests were performed to determine if the contaminated propellants would be safe to handle during the tests periods. Samples were prepared for each contaminant at each of the three concentration levels for a total of 12 samples. Ten milliliters of each sample were placed in 25-ml, low-density, polyethylene bottles and loosely capped. The samples were then stored at room temperature and observed visually throughout a two-week test period.

2.2 Shock Sensitivity Tests. The sensitivity tests were performed following the ASTM D2540-70.[4] The test uses a drop-weight apparatus to test the sensitivity of the propellant to compression ignition in a partially filled chamber. The drop-weight assembly is shown in Figure 1. In the test, 30 μl of propellant are placed in a cavity defined by an O-ring, the sample chamber, and a diaphragm. Approximately 40 μl of air are also trapped within the cavity. A 2-kg mass is dropped from a measured height onto a ball, which transmits the energy through a firing pin to the propellant cavity. A positive result is obtained if the diaphragm ruptures and the propellant is consumed. A series of tests are performed to determine the height that yields a 50 percent positive result (defined as the E50 value) with the 2-kg mass.

2.3 Closed-Bomb Tests. A closed bomb was used to obtain data on the ignition and combustion characteristics of the contaminated liquid propellants. A 30-mm regenerative liquid propellant gun (RLPG) combustion chamber was adapted to the configuration shown in Figure 2. The barrel and breech sections were replaced with plugs. The solid propellant igniter was removed, and an electrical feed-through was installed in the igniter port. The plugs were designed to give the chamber a free volume of 115 cm^3. The length-to-diameter ratio for the chamber equaled 0.25. A loading density of 0.214 g/cm^3 was used with a propellant mass of 24.6 g. The propellant was ignited using a 0.5-mm-diameter nichrome wire and an ignition voltage of 35 volts. The resistance of the wire was nominally 1.7 ohms. The wire was suspended approximately 1.0 mm below the surface of the liquid. The system was prepressurized to 3.5 MPa before firing.

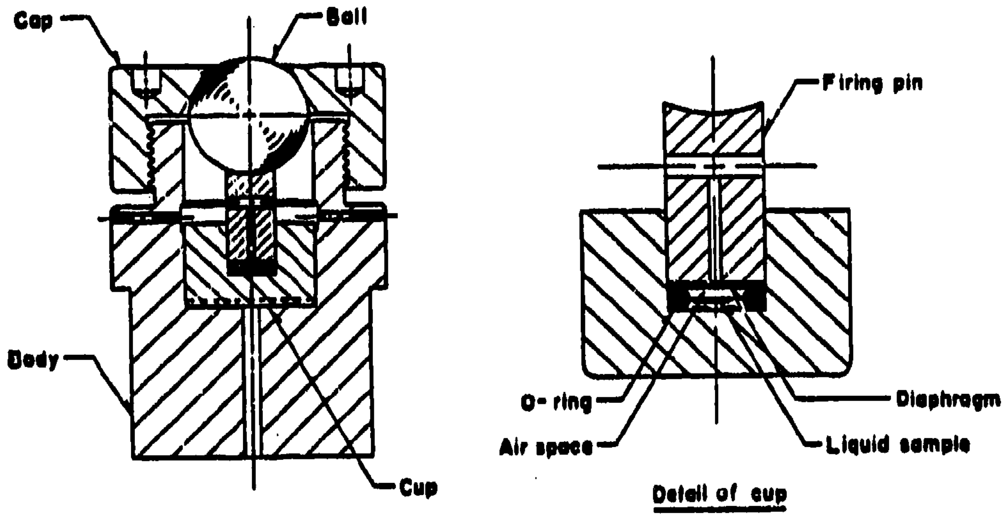

Figure 1. Drop-weight assembly.

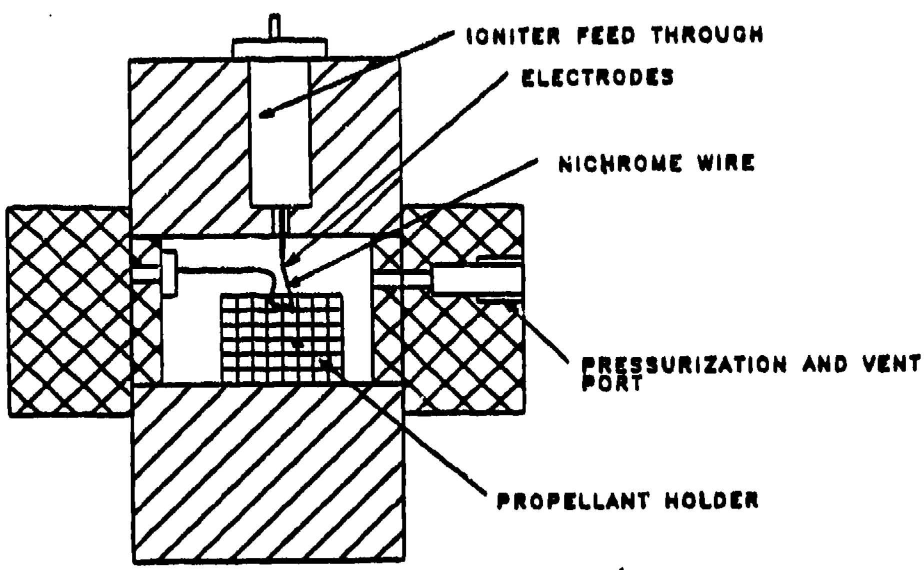

Figure 2. Closed-bomb configuration.

3. RESULTS

3.1 Storage Tests. No decompositions or fume-offs were observed during the test period with any of the samples. Of the four metal contaminants, only the iron samples showed any reaction with the propellant. All three iron concentrations showed a very small amount of gassing just after mixing. The gassing was observed as very small bubbles (less than 0.2 mm in diameter) that formed on the sides of the bottles. The gassing stopped three to five hours after the metal salt was added to the propellant. Since the amount of gassing was very small, the iron samples were tested in all later tests. The other three contaminants showed no reactions with the propellant during the storage test.

3.2 Shock-Sensitivity Tests. The results of the sensitivity tests indicate that, at ambient temperature, the contaminants did not increase the sensitivity of the propellants. At higher temperatures, all the propellant samples, including the neat propellant, increased slightly in sensitivity. The contaminants in order of sensitivity, most to least sensitive, are copper, nickel, iron, and aluminum. None of the contaminated propellant samples tested indicated possible handling problems at ambient or elevated temperatures. The results of the sensitivity tests are given below; three other materials are given for comparison:

Ambient Tests (20C +/- 1C)
(All samples at 100 ppm concentration)

LGP 1846 Neat	Insensitive
Aluminum	Insensitive up to 100 ppm
Nickel	Insensitive up to 100 ppm
Copper	Insensitive up to 100 ppm
Iron	Insensitive up to 100 ppm
Nitroglycerine	Sensitive E50 = 2 kg-cm
Normal Propylnitrate	Marginal E50 = 17 kg-cm
Hydrazine	Insensitive E50 > 100 kg-cm

High-Temperature Tests (50C +/- 10C)
(All samples at 100 ppm concentration)

LGP 1846 Neat	E50 = 100 kg-cm
Aluminum	E50 = 100 kg-cm
Nickel	E50 = 96 kg-cm
Copper	E50 = 92 kg-cm
Iron	E50 = 100 kg-cm

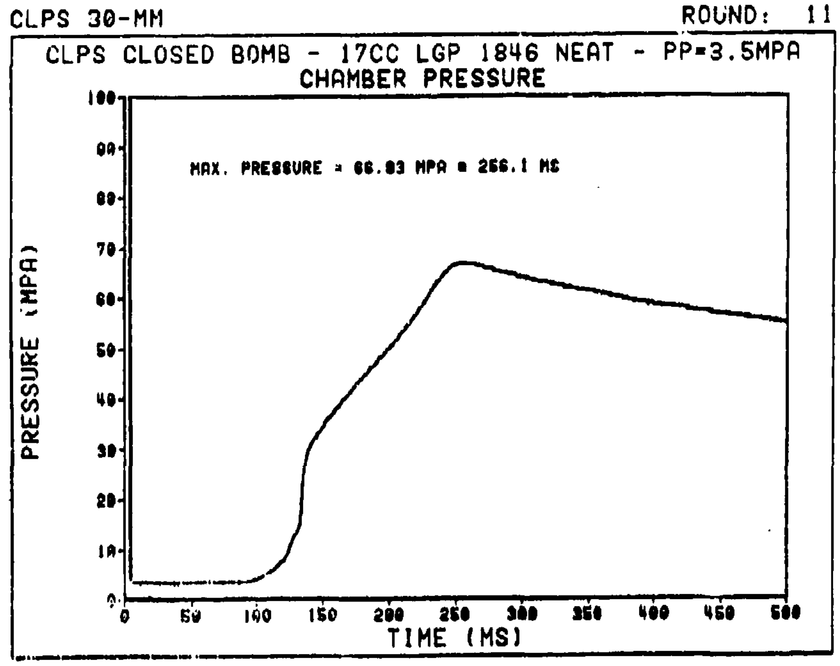

Figure 3. Pressure-time history for Test No. 11: uncontaminated LGP 1846.

3.3 Closed-Bomb Tests.

3.3.1 Observations. Figure 3 shows a complete pressure-time record from a test using uncontaminated LGP 1846. This pressure history is typical of all the tests. There is an ignition phase transitioning to rapid combustion. At approximately 35 to 40 MPa, the curve shows a sharp change in slope. This change in slope was attributed to a high heat transfer and not to the propellant.[5] Maximum pressures measured in the tests only approached 30 percent of the theoretical BLAKE pressure.[6]

A large amount of liquid residue was observed in the chamber after each test. The liquid was clear, except for black particles attributed to the burnt plastic cup, and was found to have a density nearly 1.0 g/cm^3 and a pH of approximately 5.0. The liquid is believed to be water with a small amount of unburnt fuel. Interestingly, the amount of liquid closely matched the amount of water that would be expected if all the water condensed out of the combustion products.

New thermochemical calculations[6] were made with the assumption that all water generated during combustion of the propellant condensed as it formed. This would approximate a high-heat transfer, which is typical of chambers with small L/D ratios.[5] The new, calculated value for maximum pressure closely matched the observed pressures from the experiments.

3.3.2 Tabulated Data. The results from the closed bomb firings are summarized in Table 1, and the early pressure time records for all of the tests are given in the Appendix. Listed are the maximum pressure, rise time, and pressure at onset of rapid pressure rise (transition pressure). The rise time is defined as the time for the pressure to rise from 5 MPa to 25 MPa. While this measurement is a bit arbitrary, it provides a simple method for eliminating errors in reading the actual start of pressure rise, which may be lost in the noise. The upper limit of 25 MPa was chosen because it falls in the early rapid pressure rise region, where the slope was much the same for all tests (see Figure 4).

The closed-bomb tests indicated differences in the rise times between the neat and the contaminated propellant. The neat LGP 1846 tests, nos. 11-13, showed rise times between 28.7 and 30.9 msec. The average rise time for the three tests was 30.1 msec. The propellant that was contaminated with nickel showed rise times from 22.6 to 25.1 msec. The average for the nickel tests was 23.7 msec. This gives 21 percent decrease for the rise time of the contaminated propellant compared to that of neat propellant. The aluminum contaminated propellant showed a 19.6 percent decrease. The iron and copper contaminated propellant showed 5.6 percent and 2.3 percent decreases.

In an earlier liquid-propellant, closed-bomb study, the onset of rapid pressure rise was associated with the fizz-to-flame transition of the propellant combustion.[7] As seen in Figure 4, the transition between the ignition phase and rapid-pressure rise phase is easily distinguished. There is evidence in the present data that this transition pressure is lower than that measured in the previous study. Previous values for this transition pressure were in the range of 18.3 to 22.8 MPa for LGP 1846.[7] These measurements were made by comparing the pressure record with the chamber emission. The emission was detected by a light diode mounted outside chamber. The values for the transition pressure measured during the present study, for neat LGP 1846, ranged from 15.0 to 17.8 MPa. The measurements were taken directly from the pressure-time record by observing the change in slope of the pressure rise at the transition pressure (see Figure 4). The difference between the new values and the previous values is speculated to be the delay between the actual initiation of combustion and the indication of emission by the combustion gases. Figure 5 shows a pressure record and diode history for test no. 71 of the previous study by Travis, et al.[7] The diode output fails to provide a distinct point at which combustion begins. The transition pressure for this test was 22.8 MPa. The diode output corresponding to that pressure shows that the light has already begun to increase. If one uses the pressure at which the diode first begins to indicate an increase in light intensity, the pressure is approximately 15 MPa. This number more closely agrees with the present data.

Table 1. Summary of CLPS Closed-Bomb Tests.

Round	PPM Contamination	Max pressure, MPa	Rise time for 5-25 MPa, msec	Transition pressure, MPa
11	Neat	66.8	28.7	14.4
12	Neat	66.9	30.9	15.0
13	Neat	71.5	30.8	17.8
15	75 Iron	71.7	29.3	16.6
17	86 Iron	65.0	27.4	16.3
18	86 Iron	72.1	28.6	16.4
19	112 Copper	67.5	29.1	-----
20	112 Copper	69.9	31.4	16.4
21	112 Copper	70.2	27.6	15.2
23	94 Nickel	69.4	25.1	15.9
24	94 Nickel	68.4	23.4	13.8
25	94 Nickel	70.4	22.6	13.8
26	83 Aluminum	71.0	24.4	15.7
27	83 Aluminum	71.1	26.7	15.1
28	83 Aluminum	69.2	21.6	-----

NOTE: Initial conditions: propressure = 3.5 MPa;
Loading density = 0.214 g/cm^3; and
LP volume = 17.0 cm^3.

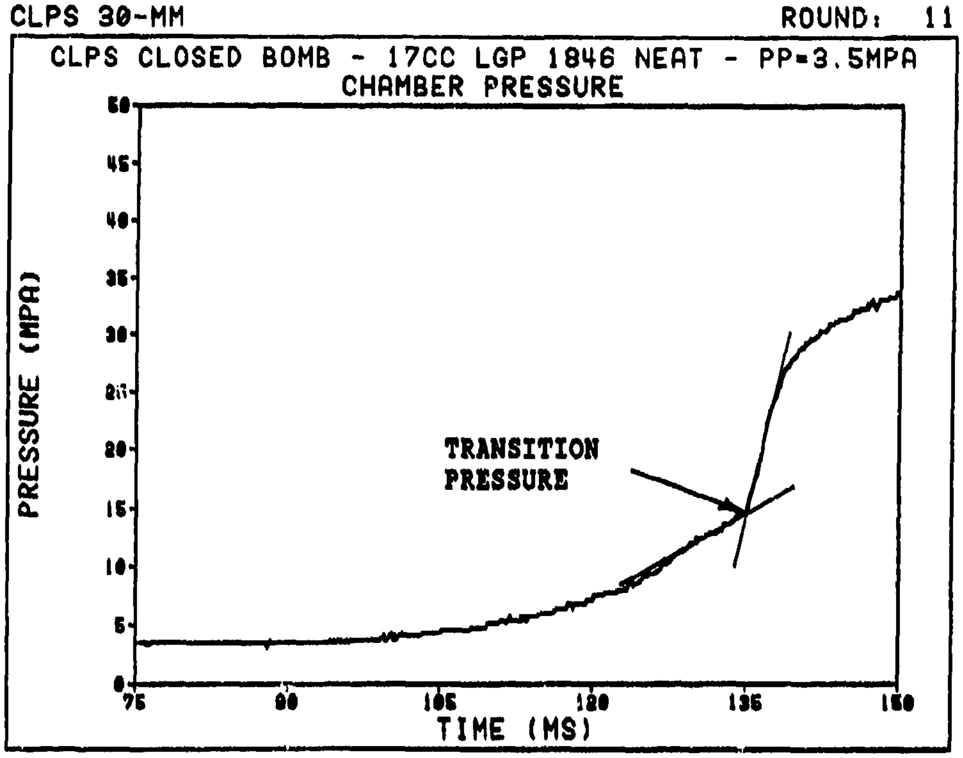

Figure 4. Transition pressure for Test No. 11.

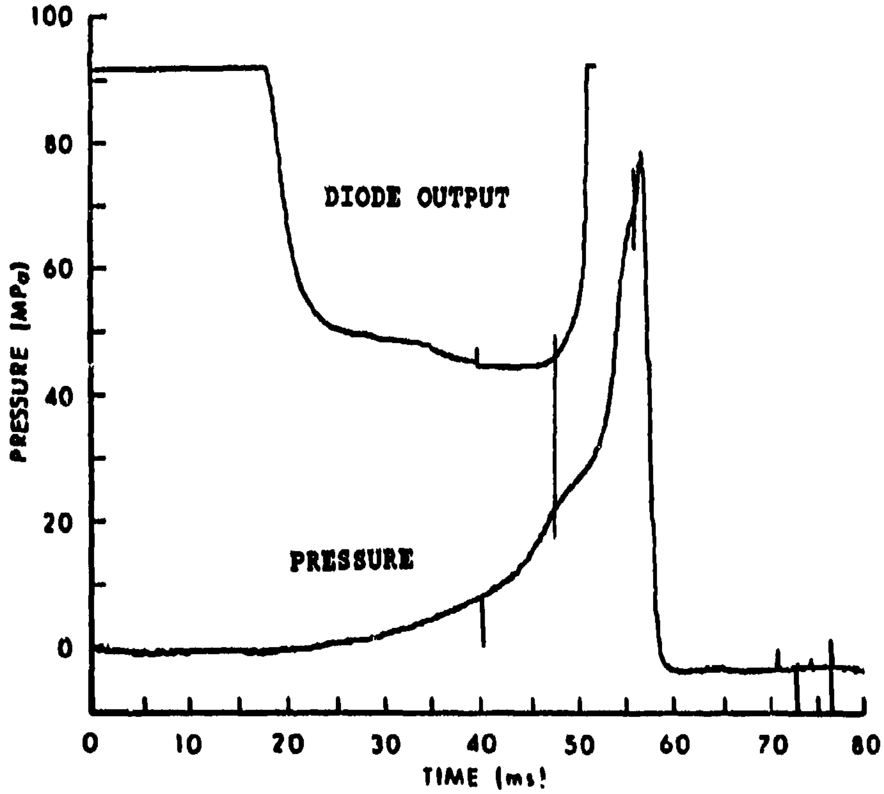

Figure 5. Pressure-time and diode output history for Test No. 266-71.[7]

4. DISCUSSIONS AND CONCLUSIONS

Four metals—iron, copper, nickel, and aluminum—were introduced into LGP 1846 as contaminants. These contaminated propellants were then subjected to three types of tests: storage tests, shock-sensitivity tests, and closed-bomb tests.

All four contaminated samples appeared safe at concentration levels of less than 100 ppm for the duration of the storage test, i.e. two weeks. The iron samples produced very small bubbles that did not present a problem during the later tests.

The shock sensitivity drop weight tests indicated that the presence of metal contaminants will increase the sensitivity of LGP 1846 at higher temperatures. The increase was small, and the propellant remained in the safe regime.

The most significant result of the closed bomb tests was the decrease in rise times of the contaminated propellants compared to those of the neat propellant. The largest decrease in rise time was associated with the nickel and aluminum contaminants. The iron and copper contaminants provided only small decreases in rise times. The rapid combustion characteristics (at pressures above the fizz-to-flame pressure) did not appear affected by the contaminants.

The fizz-to-flame or transition pressure of the LGP 1846 in this study is lower than previously reported. The values of the transition pressure for neat LGP 1846 ranged from 14.4 to 17.8 MPa. The transition pressure for the propellants that were contaminated did not differ greatly from that for the neat propellant, indicating that the contaminants did not have a significant effect on the fizz-to-flame transition of the HAN-based propellants.

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APPENDIX :

PRESSURE-TIME RECORDS FOR TESTS WITH METAL CONTAMINANTS

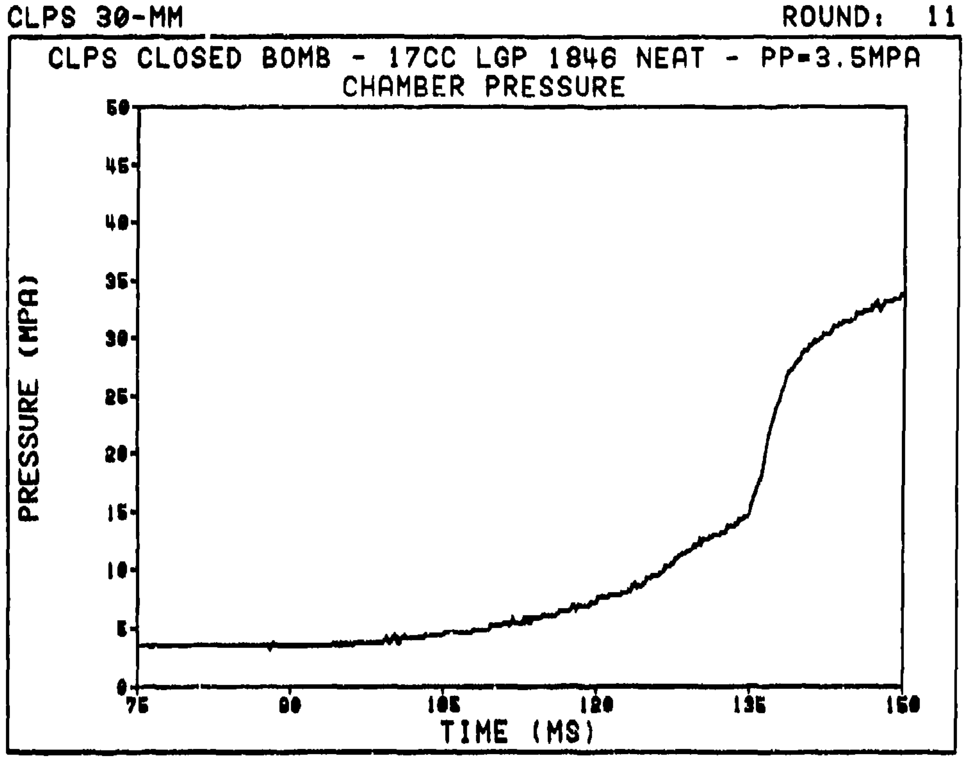

Figure A1. Pressure record from round 11; no contamination; LGP 1846.

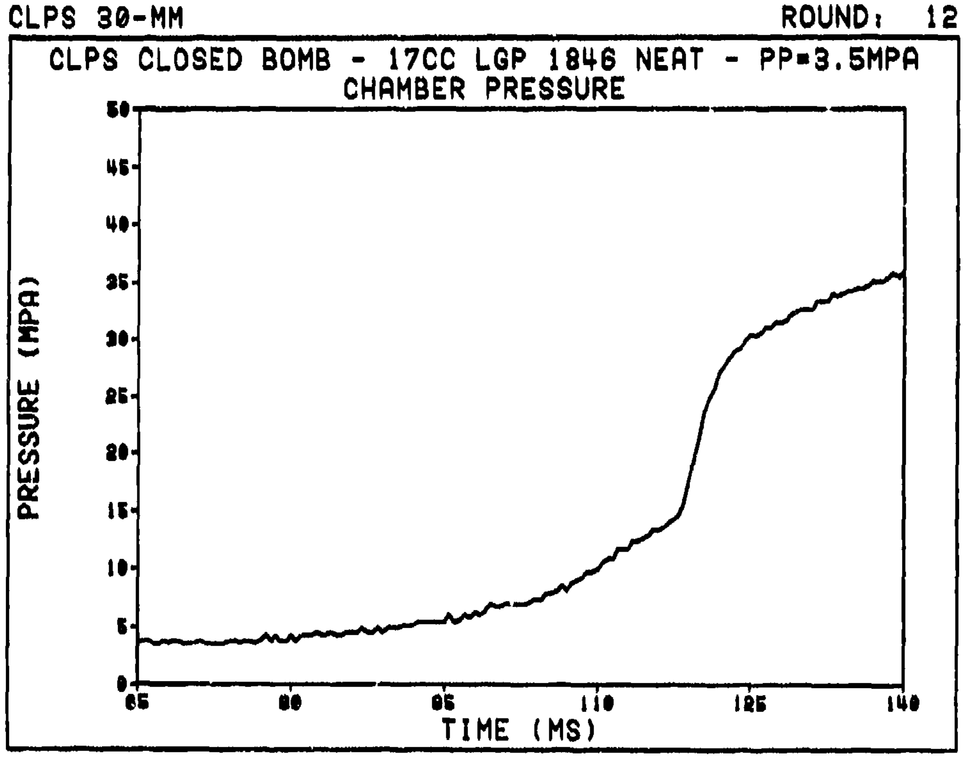

Figure A2. Pressure record from round 12; no contamination; LGP 1846.

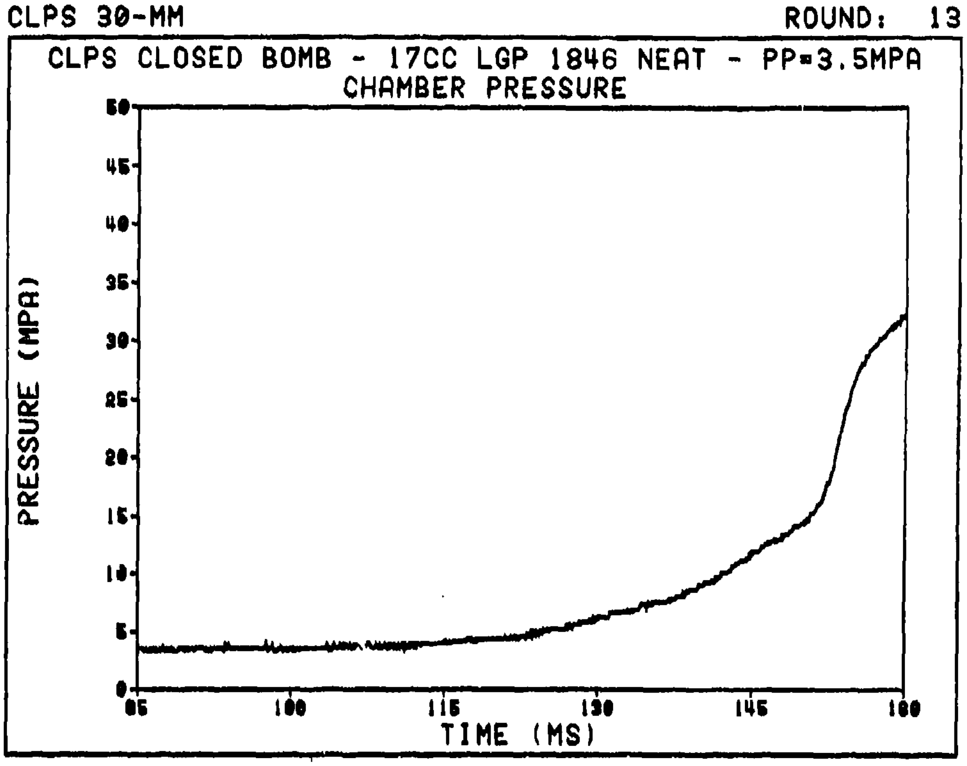

Figure A3. Pressure record from round 13; no contamination; LGP 1846.

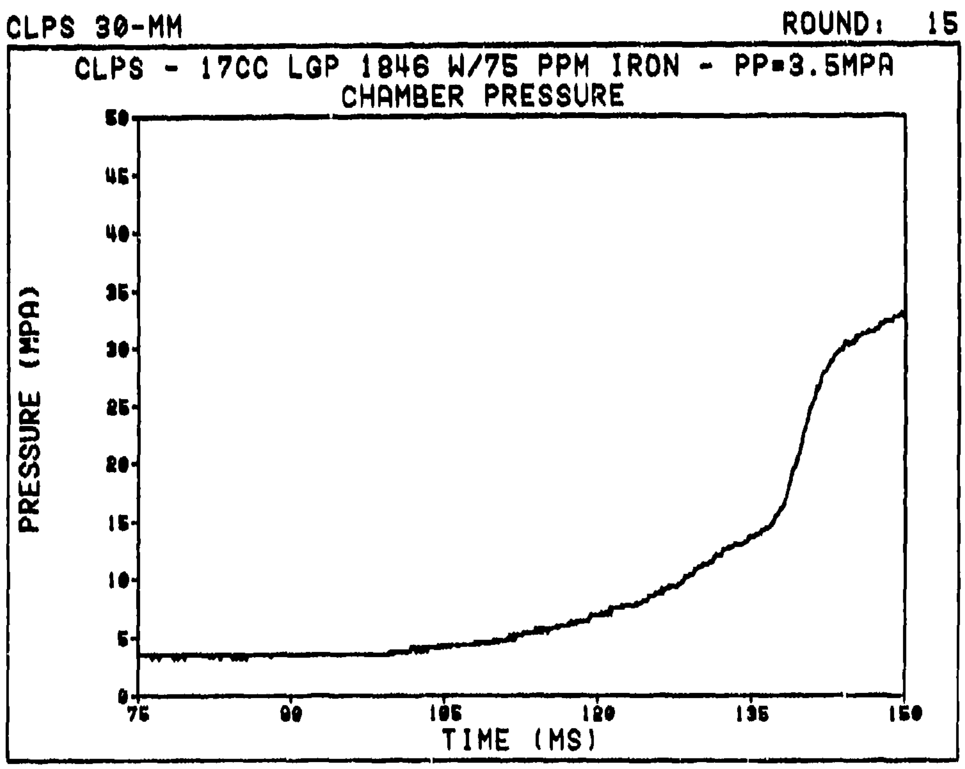

Figure A4. Pressure record from round 15-75 ppm iron; LGP 1846.

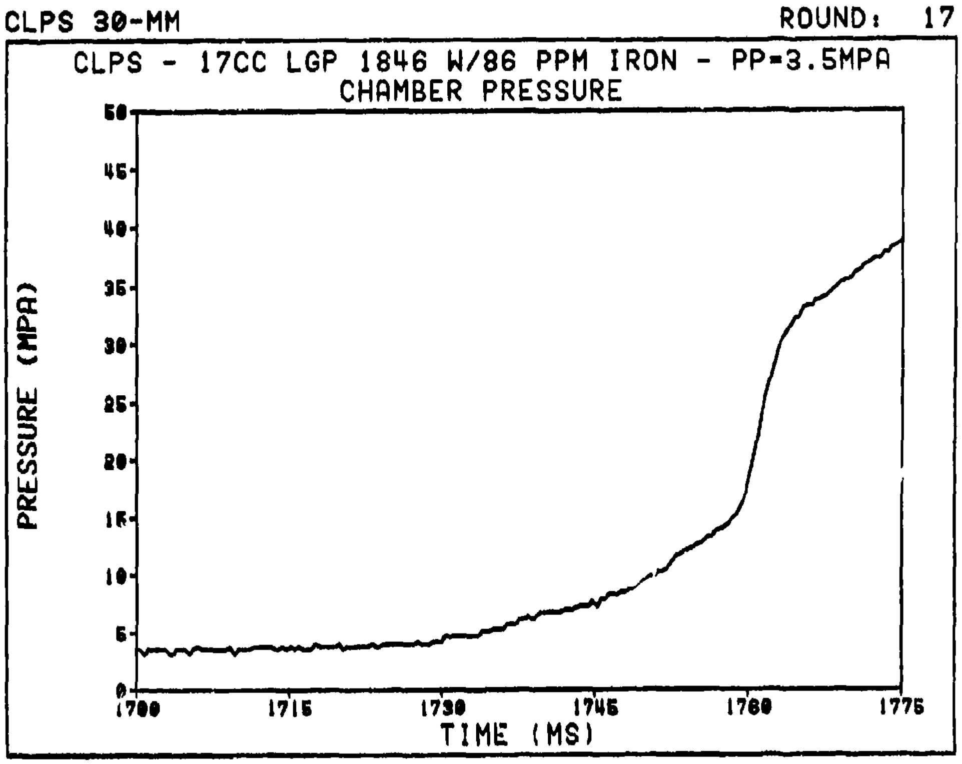

Figure A5. Pressure record from round 17-86 ppm iron: LGP 1846.

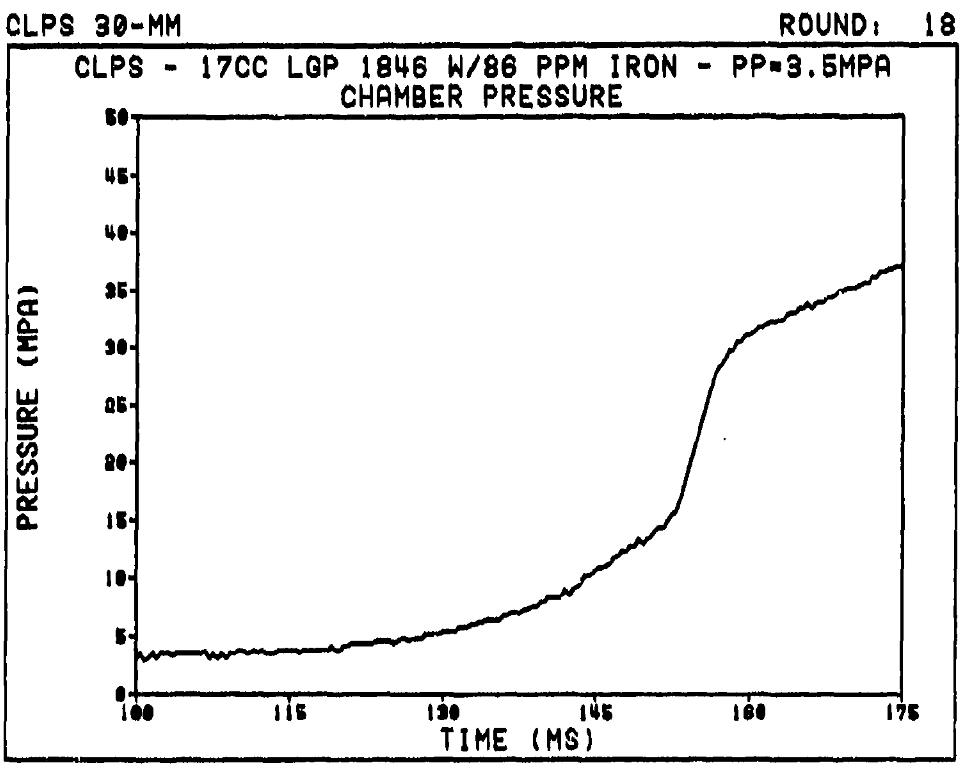

Figure A6. Pressure record from round 18-86 ppm iron: LGP 1846.

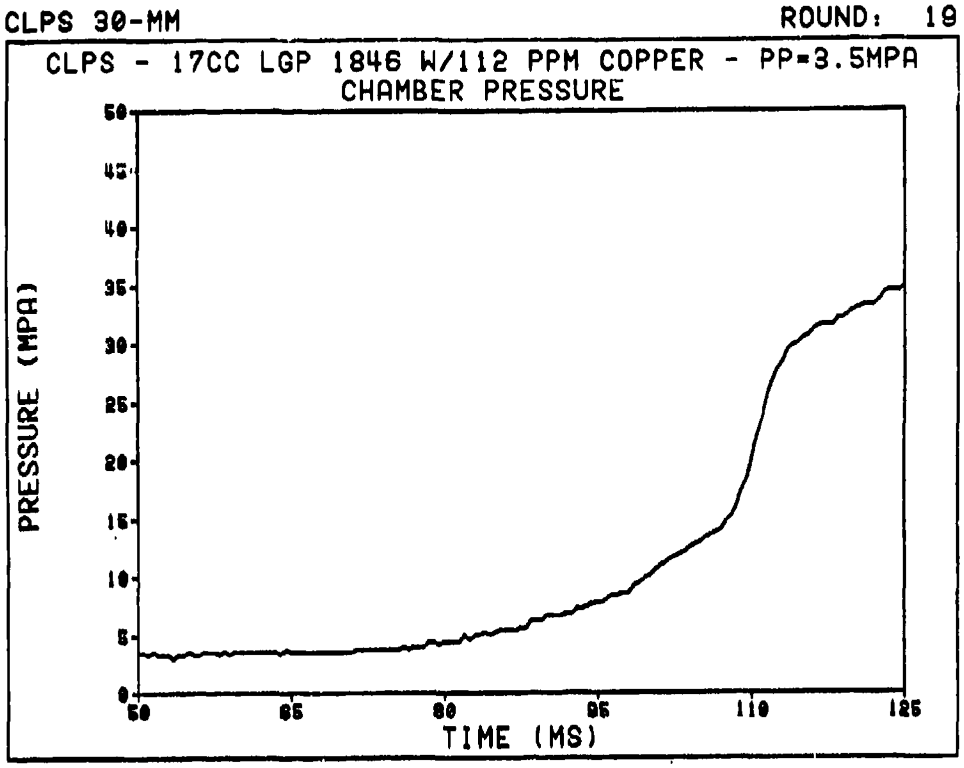

Figure A7. Pressure record from round 19-112 ppm copper; LGP 1846.

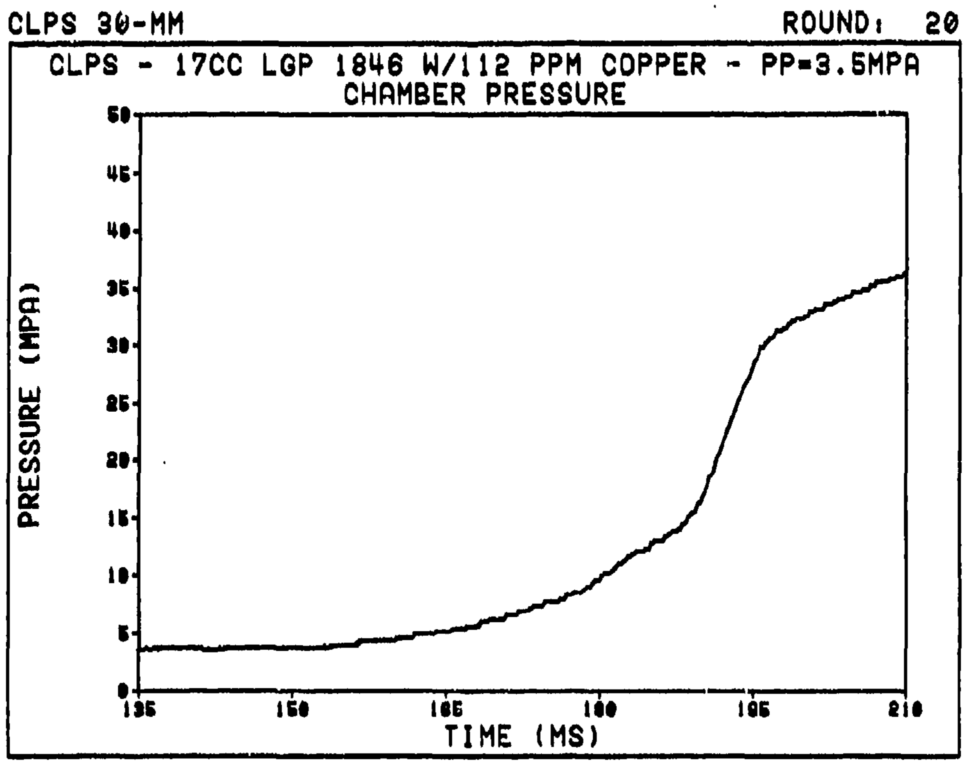

Figure A8. Pressure record from round 20-112 ppm copper; LGP 1846.

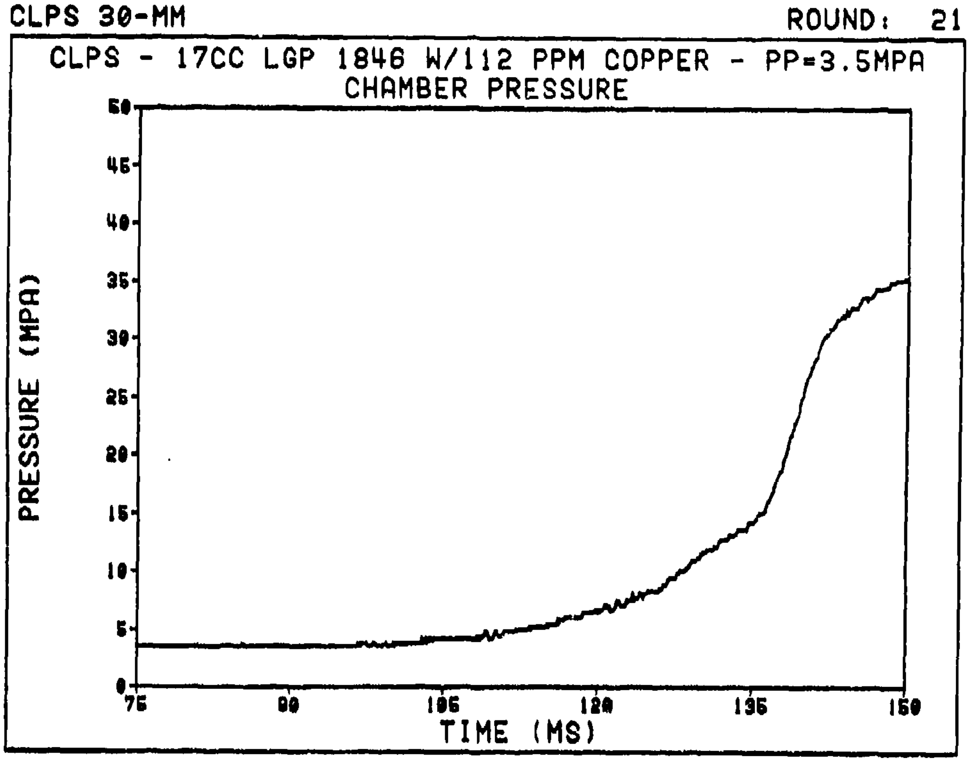

Figure A9. Pressure record from round 21-112 ppm copper: LGP 1846.

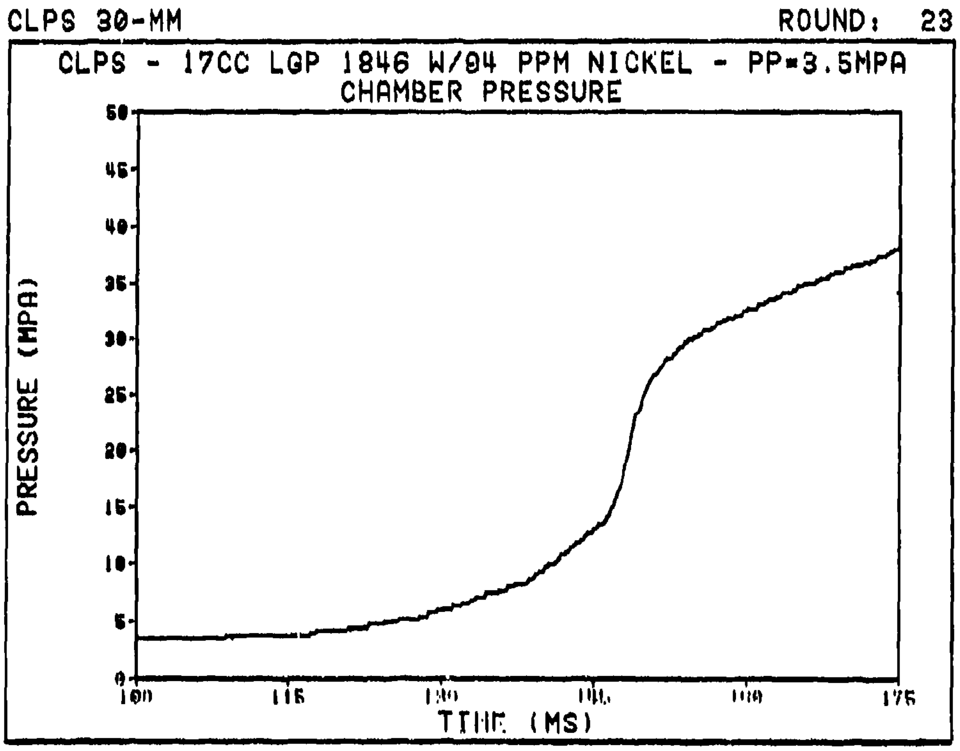

Figure A10. Pressure record from round 23-94 ppm nickel: LGP 1846.

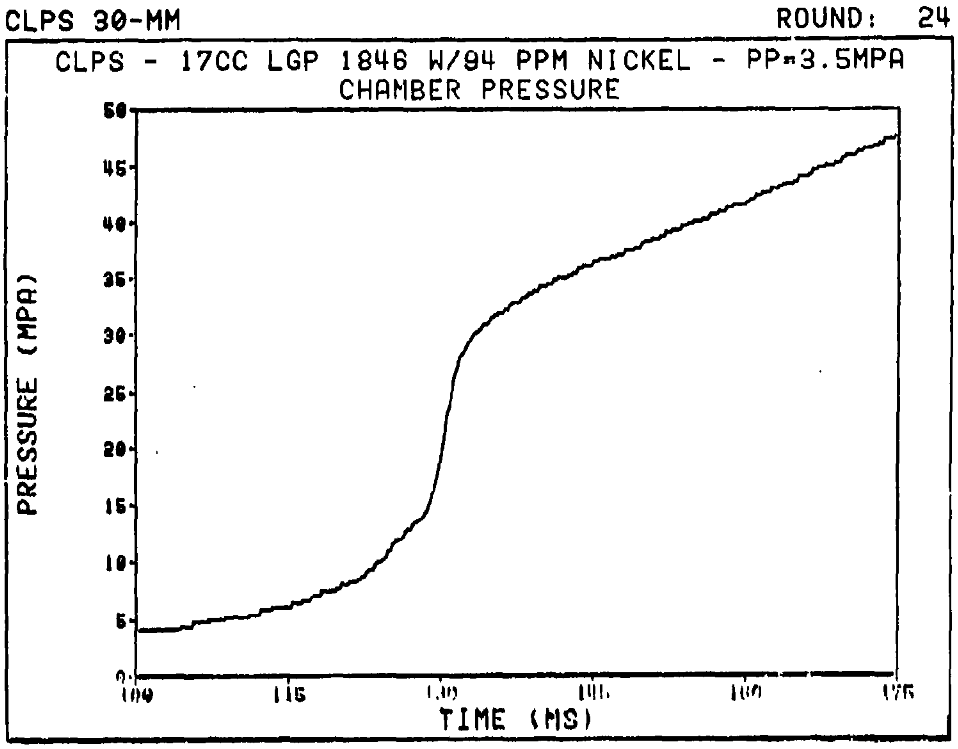

Figure A11. Pressure record from round 24-94 ppm nickel; LGP 1846.

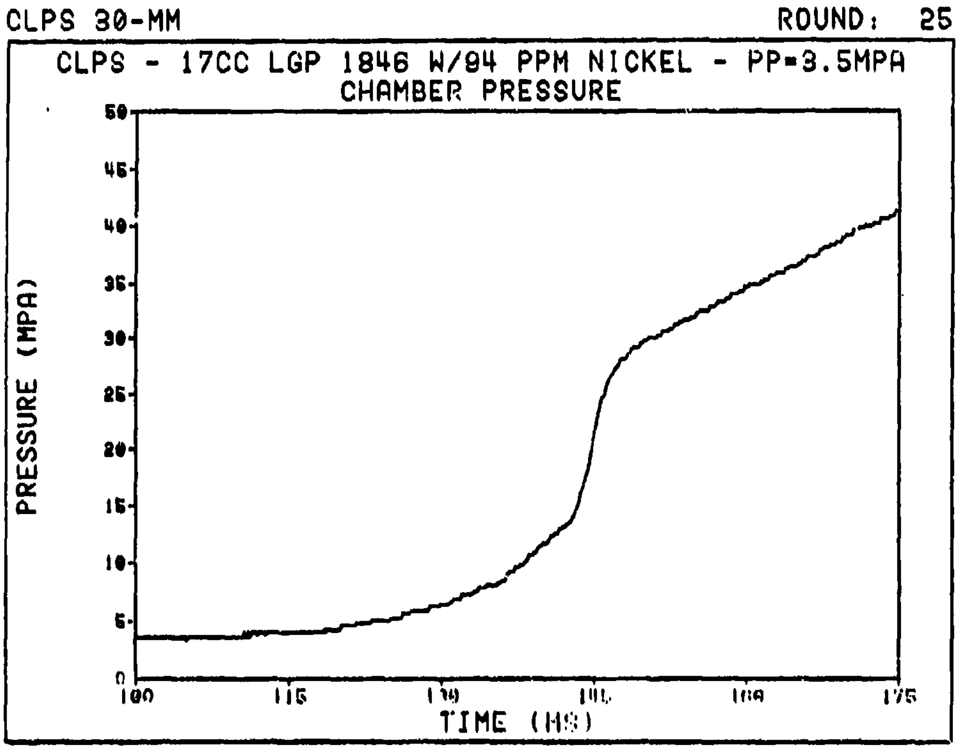

Figure A12. Pressure record from round 25-94 ppm nickel; LGP 1846.

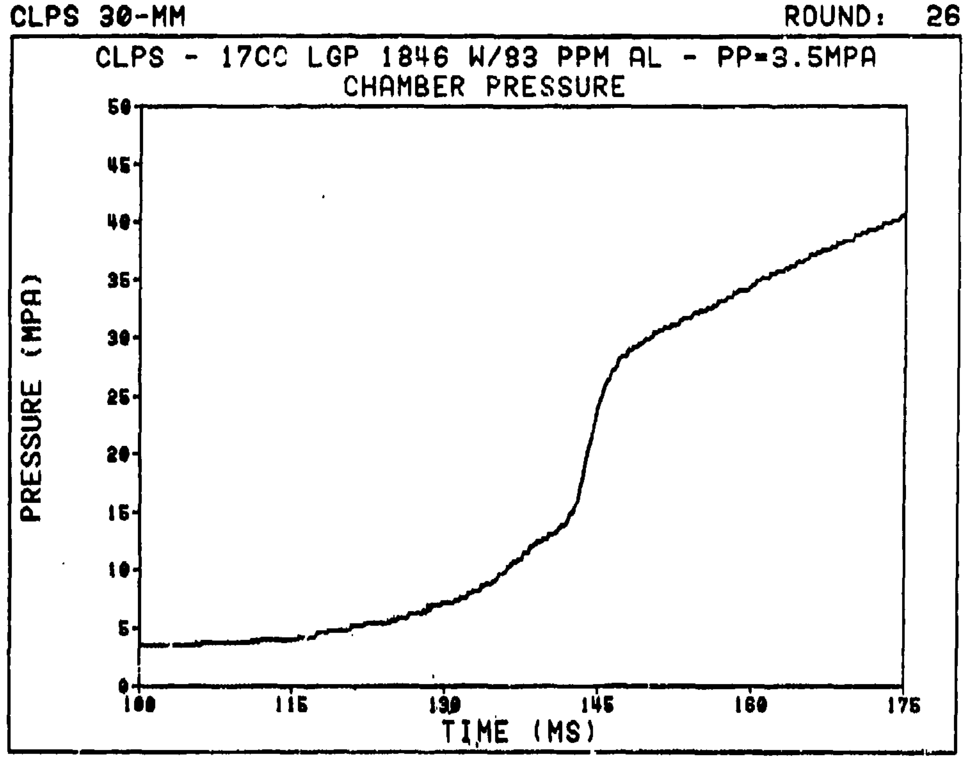

Figure A13. Pressure record from round 26-83 ppm aluminum: LGP 1846.

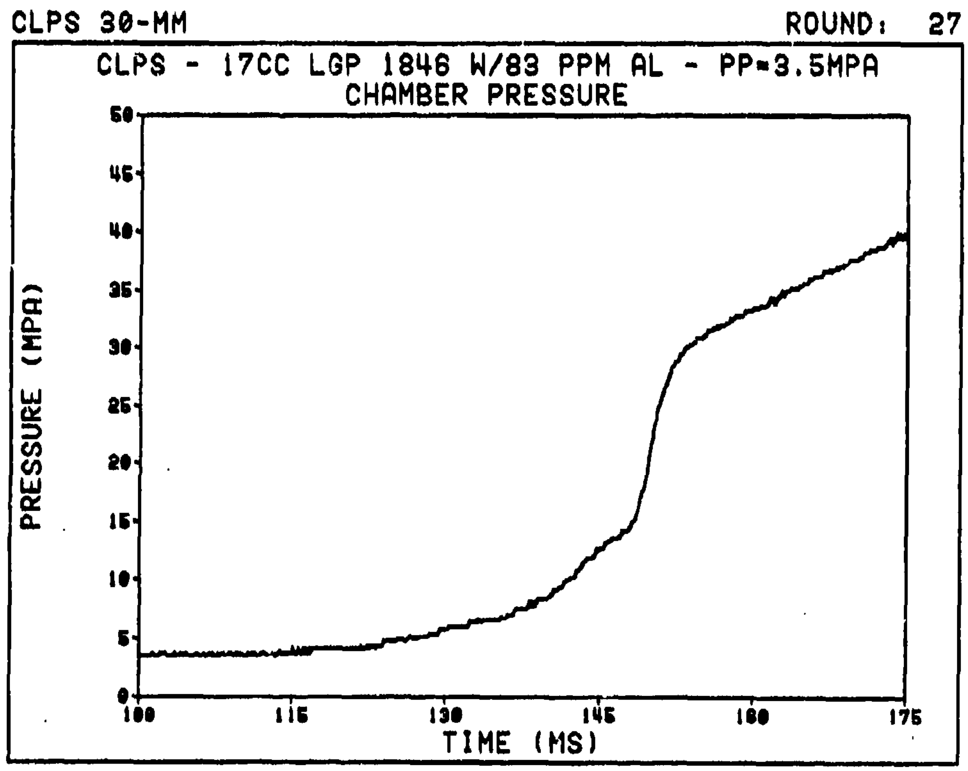

Figure A14. Pressure record from round 27-83 ppm aluminum: LGP 1846.

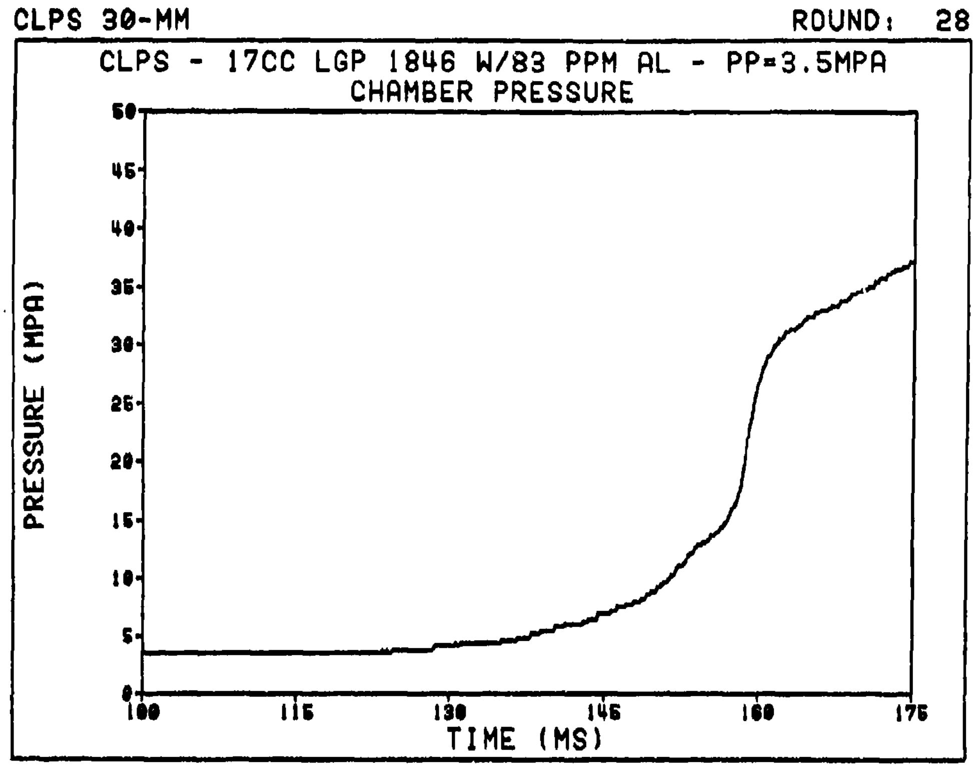

Figure A15. Pressure Record from Round 28-83 ppm aluminum; LGP 1846.

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